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TECHNICAL MEMORANDUM 2011

STUDY ON SYNTHESIS

OF

N - NITROBORAZINE COMPOUNDS

II. BORAZINE DERIVATIVES

BY

T. HIRATA

JUNE 1971

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Propellants Laboratory
Feltman Research Laboratories
Picatinny Arsenal
Dover, New Jersey

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13. ABSTRACT

Attempts to synthesize borazine derivatives for use in propellants and related tems are described. Borazine itself was unsuitable as starting material since it has a tendency to polymerize and/or decompose at room temperature; however, B-trimethylborazine is extremely stable from decomposition or attack from air or moisture. Borazine reacted violently with nitryl chloride in the absence of diluent. In solution, the reaction was moderated but vigorous and the borazine appeared to have been completely oxidized. Synthesis and isolation of B-trimethylborazine from B-trichloroborazine and methyl lithium or methyl magnesium chloride proved to be extremely tedious. In addition the yields were much lower than expected. Conditions were varied but yields did not improve.

The regeneration of borazine from its solid complex with hydrogen chloride was investigated in order to devise a convenient method for its storage.

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N-nitroborazines						
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Borazine - HNO_3 complex						
B-trichloroborazine						
Storage of borazine						

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ABSTRACT

Attempts to synthesize borazine derivatives for use in propellants and related items are described. Borazine reacted violently with nitryl chloride in the absence of diluent. In solution, the reaction was moderated but vigorous and the borazine appeared to have been completely oxidized.

Since borazine has a tendency to decompose and/or polymerize at room temperature, the use of B-trimethylborazine with its superior moisture and thermal stabilities was judged to be more amenable to experimentation. Synthesis and isolation of B-trimethylborazine from the reactions of B-trichloroborazine with methyl lithium or methyl magnesium chloride proved to be extremely tedious. Although conditions were varied, the yields were disappointingly lower than expected.

The problem of storing borazine as the solid complex with hydrogen chloride was explored, particularly with regard to its regeneration. The possibility of convenient storage was apparent but the efficiency of regeneration required further improvement. The borazine complex with hydrogen nitrate was also examined.

CONCLUSION

Borazine reacts violently with nitryl chloride in the absence of diluent. In solution the reaction is moderated but still vigorous. Decomposition of the molecule rather than displacement on the N-H hydrogen to form N-nitroborazine occurs. Thus the usual nitration techniques will not be applicable to synthesize nitroborazine derivatives. It was not determined whether N-nitro-B-alkylborazines can be synthesized through alternate routes. The fact that dimethylnitramine was obtained in the reaction between lithium dimethylamide and nitryl chloride, as described in Part I, PATM 2010, indicates that the synthesis of N-nitroborazines may be possible via the reaction of N-lithium borazines and nitryl chloride.

An obvious additional reaction which should be carried out is that between boron trichloride and nitramide itself in the same manner as the reaction between boron trichloride and ammonium chloride. This reaction may lead directly to B-trichloro-N-trinitro borazine from which additional derivatives may be obtained.

The solid complex of borazine with hydrogen chloride should be further investigated with the objective of using this as a means of storage since borazine slowly decomposes even at room temperature and therefore cannot be kept on the shelf for extended lengths of time.

A critical examination of the borazine complex with hydrogen nitrate should be made to verify its existence or non-existence.

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INTRODUCTION

Although the organo-nitramines have long been utilized in pro-
pellants and explosives, the boron nitramines are unknown and have
not been investigated to date. Borazine is a six membered cyclic com-
pound of alternating boron and nitrogen atoms, isoelectronic with
benzene:
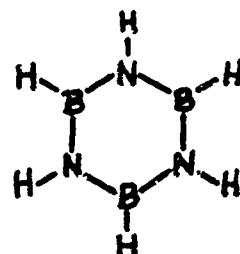
Derivatives of borazine with substitutents on nitrogen are normally
synthesized by reacting substituted amines with the appropriate boron
derivatives, while substituents on boron can be obtained either by
starting with substituted boron dichlorides or by reacting borazine
and/or B,B',B"-trichloroborazine with lithium alkyls or with Grignard
reagents. Of the substituted borazines, those with organic substituents
predominate and the number of borazine derivatives with inorganic
substituents are limited. The chloro- and amino- borazines are ex-
amples of the inorganic class of derivatives. As yet, the N-substituted
nitroborazines have not been reported nor does any work appear to have
been directed toward the synthesis of such compounds. N-nitro, N,N',N"-
trinitro borazine are hypothetical compounds not only of interest from
a synthetic standpoint but also from the standpoint of generating a new
family of propellants and related items incorporating boron.

The objective of this program was to synthesize N,N'N"-trinitro-
borazine, a hypothetical compound analogous to cyclotrimethylenetri-
nitramine (RDX), where the tetracovalent carbons are replaced by tri-
valent borons. Since the boron attains a sextet of electrons in borazine
leaving an unoccupied orbital while nitrogen has an unshared pair of
electrons, resonance is possible in the ring from electron delocalization.
Resonance effects in borazine have been extensively studied (Ref. 1).
In contrast to RDX where the saturated carbon and nitrogen atoms prevent
resonance in the ring, resonance is possible in symmetrical trinitro-
benzene with which N-trinitroborazine* is isoelectronic. Thus from a

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* The incorrect but accepted method of designating the di- and tri-
substitutions simply as N-dinitro- and N-trinitro borazine instead of
N, N'-dinitro- and N, N', N"-trinitroborazine will be used henceforth.
B-substituted borazines will also be similarly designated.

theoretical consideration, the synthesis of N-nitroborazine derivatives presents a challenge and a possibility of further examining the "benzene" characteristics of cyclic B-N compounds. From a practical standpoint, moreover, this would be a method of obtaining a propellant incorporating boron.

One of the drawbacks of both RDX and HMX oxidizers is the fact that no method has yet been found to replace the unreactive methylene hydrogen atoms with substituents. On the other hand, there is an abundance of B-substituted borazines and thus the number of possible B-substituted N-nitroborazines is almost unlimited. The possibility of replacing the hydrogen attached to boron introduces a greater flexibility to fit nitramine oxidizers to a given set of requirements such as incorporation into polymers or as crosslinking agents containing nitro groups.

Early in the study it was found that borazine itself did not have the desired thermal stability since decomposition occurs even at room temperature and it is susceptible to attack by moisture and/or oxygen from the atmosphere. Furthermore, the B-H rather than the N-H bond is preferentially attacked in substitution reactions and therefore reactivity of the borons toward entering groups must be neutralized in order to effect substitution on the nitrogen atoms. The thermal stability of B-trimethylborazine has been found to be higher than that of triethylborazine and borazine itself (Ref. 2). In addition to the electron releasing inductive effect, with boron atoms blocked by methyl groups, it was expected that substitution reactions would be directed toward the nitrogen atoms of the ring. Thus the immediate objective of this study was to obtain B-trimethyl-N-trinitroborazine, a compound isoelectronic with trinitromesitylene.

Although various nitrating routes were considered, a metathetical reaction of nitryl chloride (NO_2Cl) with N-lithium borazine derivatives was expected to give the desired products.

The pathway to obtain the proposed compound was to first synthesize B-trimethylborazine after determining the best of several synthetic routes. Then, synthesize the intermediate B-trimethyl-N-trilithium borazine from which the target compound was to be obtained in a metathetical reaction with nitryl chloride:

$$B_3(CH_3)_3N_3H_3 + LiR \longrightarrow B_3(CH_3)_3N_3Li_3 + 3 RH$$

followed by:

$$B_3(CH_3)N_3Li_3 + 3\ ClNO_2 \longrightarrow B_3(CH_3)_3N_3(NO_2)_3 + 3\ LiCl$$
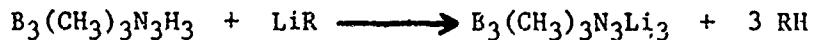
Thus several intermediate syntheses were required and the only reaction which had not been reported in the literature was the formation of nitramines from nitryl chloride and lithium amides:

$$R_2N\text{-}Li + ClNO_2 \longrightarrow R_2N\text{-}NO_2 + LiCl$$
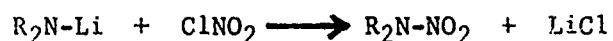
The reaction of nitryl chloride with lithium dimethylamide is described elsewhere (Part 1, PATM 2010).

RESULTS AND DISCUSSION

1. Reaction of Borazine with Nitryl Chloride

The reaction of 1.5 moles of neat borazine with excess nitryl chloride was sufficiently violent to destroy a 50 ml round bottom flask reactor as well as the Dewar flask which enclosed the reactor. In tetrahydrofuran (THF), the reaction was moderated but still vigorous. Bright orange and red color changes accompanied the progress of reaction. Borazine was completely consumed leaving a brown-yellow amorphous solid which bleached white in air and did not show a melting point up to 180°C. The infrared spectrum of the solid was similar to that of the crystalline material obtained from the hydrolysis of the viscous liquid isolated from borazine (discussed under "Borazine" in the Experimental Section). The reaction apparently was an oxidation of borazine rather than displacement on the hydrogens of the ring.

2. Reaction of Borazine with Gaseous Hydrogen Chloride

Since liquid borazine is known to decompose even at ambient temperature (Ref. 3) it cannot be stored at room temperature for extended periods. However, it reacts with simple halogen acids to form stable white solid complexes. Thus, a re-examination of the borazine complex with HCl was made in order to ascertain the possibility of a convenient means of storage.

Borazine reacts with gaseous HCl and HBr in a molar ratio of 1:3 to form a white solid from which borazine regeneration is apparently possible upon heating. Laubengayer, et al (Ref. 4), carried out a detailed investigation of the halogen acid-borazine complex but had not considered the possibility of borazine regeneration. The infrared absorption spectrum of the solid complex indicated the presence of an

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N-H doublet around 3200 cm^{-1}, a B-H singlet around 2500 cm^{-1} and a B-Cl singlet at 1032 cm^{-1}. However, because of its amorphous character, insolubility in several organic solvents and the absence of the characteristic borazine ring vibration frequency around 1400 cm^{-1}, it was concluded that the material may not have been a simple ring system but may have been polymeric. According to their pyrolysis and thermogravimetric data, borazine recovery from the solid complex upon thermal treatment was nil while approximately 60% was the complex itself which had reformed in a cold trap and between 10-20% was non-volatile residue, presumably p lymerized material. On the other hand, mass spectroscopic data from the pyrolysis at 110°C of the borazine-3HBr complex indicated that approximately 80% of the volatile species was borazine.

Dahl and Schaeffer (Ref. 5) in deuterium isotope exchange studies of borazine reported a recovery of approximately 30% borazine from the borazine-3DCl complex by heating the complex at 70-80°C in the presence of triethylamine.

Thus whether the complex maintains the monomeric integrity of a cyclic structure or polymerizes, dehydrohalogenation at moderate temperatures (less than 100°C) in the presence of a base regenerates borazine.

Figure 1 is an infrared spectrum of the borazine-3HCl complex made by the KBr pellet technique. The absorption peaks around 3200^{-1}, 2500 cm^{-1} and 1020 cm^{-1} correspond to those reported by Laubengayer, et al (Ref. 4). In addition, the broad absorption band centered at 1400^{-1}, which they did not observe, could be the characteristic ring distortion frequency of the borazine ring. This frequency ranges from 1392 cm^{-1} in B-trichloro-N-trimethylborazine to 1482 cm^{-1} in B-trimethylborazine (Ref. 6). This is normally the most intense peak in borazine and its derivatives. Thus its medium intensity as well as its breadth (Fig. 1) indicate that the structure of the borazine-3HCl complex has been considerably altered. X-ray diffraction data for the complex are also given in Table 1. The data indicate that more than one compound was present. One belonging to a cubic system and a second component probably arising from hydrolysis when the sample was exposed to the atmosphere while filling a capillary tube for X-ray analysis.

The simple addition of trimethylamine to a THF solution of the borazine-3HCl complex did not displace HCl and release borazine. Either the nitrogen in the borazine ring is a stronger base than trimethylamine, or the formation of a cyclic, substituted ammonium salt where nitrogen attains the tetracovalency of the ammonium ion with the two adjacent borons of ring and two hydrogens:

$$\left[\begin{array}{c} B \\ \\ B \end{array}\!\!\!\!>\!\!N\!\!<\!\!\!\!\begin{array}{c} H \\ \\ H \end{array}\right]^{+}$$
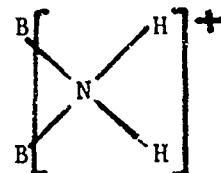
while boron attains the tetracovalency of the borohydride or tetra-
chloroborate anion with two adjacent nitrogens, hydrogen and chlorine:

$$\left[\begin{array}{c} N \\ \\ N \end{array}\!\!\!\!>\!\!B\!\!<\!\!\!\!\begin{array}{c} H \\ \\ Cl \end{array}\right]^{-}$$
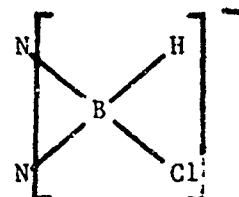
produces a thermodynamically stable configuration which requires con-
siderable energy to return the nitrogens and borons to their original
trivalent state.

However, under appropriate conditions, borazine can be regenerated
from the solid. For example in another experiment, approximately six cc
of triethylamine previously dried over barium oxide and degassed was
condensed on two millimoles of the solid borazine-3HCl complex. The
mixture was heated with a hot water bath at 50°C for five hours and at
72°C for an additional two hours. An infrared spectrum of the gaseous
products proved to be that of the amine and no absorption peak attribut-
able to borazine was present; but when the complex and triethylamine was
heated again at about 75°C for three hours a definite borazine absorption
spectrum was obtained from the gaseous products. Although the borazine
could not be isolated from the amine by simple low temperature fraction-
ation, when sufficient boron trifluoride was added to form a complex
with the amine, a mixture of borazine and boron trifluoride was re-
covered as a fraction which was volatile at -78°C. Borazine was easily
isolated at -110°C (carbon disulfide slush bath) where boron trifluoride
still has considerable pressure. Recovery of borazine was approximately
ten percent.

No recovery of borazine resulted when the complex was heated be-
tween 75-80°C in the presence of pyridine.

Base strength, temperature and solvent are important variables to
consider in the regeneration of borazine. Too high a temperature can
decompose the borazine being regenerated and the choice of solvent can
determine whether the borazine can be easily isolated. The base strength
or the type of base is of prime importance since the displacement of HCl
is necessary, but the borazine-3HCl complex is also capable of reaction
resembling ring substitution. Dahl and Schaeffer (Ref. 7) were able

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to displace chlorine with hydrogen by means of the reaction between sodium borohydride and borazine-3HCl complex in diglyme at room temperature and obtained hexahydrocycloborazane:

$$3B_3N_3H_6 \cdot 3HCl + 6NaBH_4 \longrightarrow 2B_3N_3H_{12} + 6NaCl + 3B_2H_6$$
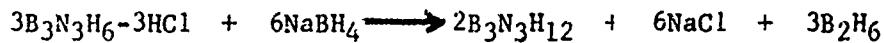
Although the hexahydrocycloborazane yield was not reported, a 75% yield of diborane according to the above equation was obtained. This indicated that the borazine-3HCl complex is a cyclic substituted ammonium salt structurally similar to cyclohexane:
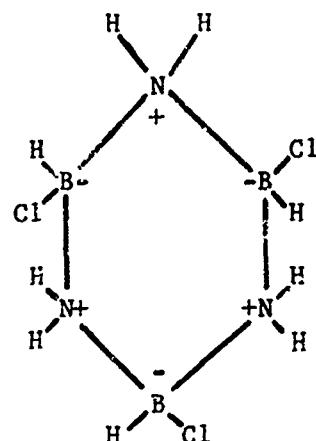
Thus the driving force in the reaction with sodium borohydride is the formation of NaCl and B_2H_6, whereas the reaction in the presence of triethylamine is thermal dehydrohalogenation followed by the Lewis acid-base reaction to form amine hydrochloride which prevents recondensation of the borazine complex.

To date, this complex of borazine has been inadequately examined. In the present study sufficient variation of variable were not attempted and definitive information with regard to its characteristics is lacking. The fact that mass spectroscopic data indicated that the volatile species at 110°C from the borazine-3HBr complex was 80% borazine is also good indication that borazine could be more conveniently stored and regenerated.

## 3. Attempt to Form the Hydrogen Nitrate Complex of Borazine

Addition of silver nitrate in acetonitrile to a solution of the borazine-3HCl complex in toluene immediately formed a cloudy white precipitate. Upon prolonged standing, a small amount of colorless needle crystals were obtained. The material did not melt up to 200°C. it was soluble in water and qualitative tests on the solution indicated presence of nitrate and absence of silver and chloride ions. Flame

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test on a crystal was green, characteristic of boron. Crude tests indicated that the material was soluble in acetone, somewhat soluble in THF and diglyme (diethylene glycol dimethyl ether) and insoluble in toluene or chlorobenzene. No further tests to characterize the material was made due to the insufficiency of sample. Since silver and chlorine were absent and only boron and nitrate were present, a tentative assumption is that the borazine-3HNO$_3$ complex had formed; however, assuming that the HNO$_3$ complex would be similar to the HCl complex, its crystallinity and insolubility in THF and diglyme in contrast to the HCl complex would seem to indicate otherwise.

When borazine itself was reacted with silver nitrate in acetonitrile, the solution turned yellow, then orange and finally a dark amorphous mass remained indicating that decomposition of borazine had occurred. Thus it appears that in the reaction of silver nitrate with the borazine-3HCl complex, the chloride was eliminated as silver chloride but the borazine entity was then oxidized by the nitrate which would indicate that the borazine-3HNO$_3$ complex is unstable with respect to oxidation.

4. Reactions of B-Trichloroborazine

The reactions of Grignard and organo-lithium reagents with B-trichloroborazine or with N-substituted borazines have been used to obtain B-alkylborazine derivatives (Refs. 2 & 8).

$$B_3Cl_3N_3H_3 + 3RMgX \longrightarrow B_3R_3N_3H_3 + MgClX$$

$$B_3H_3N_3R_3 + 3RLi \longrightarrow B_3R_3N_3R_3 + LiH$$
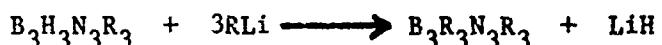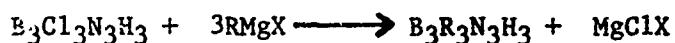
Although milligram quantities of B-trimethylborazine were synthesized, scaling-up to gram quantities met with little success. The choice between the Grignard reaction or the alkyl lithium reaction is primarily determined by the availability of reagents. The Grignard reaction is perhaps more convenient since the reagent can be made in-situ by the addition of methyl iodide to a mixture of magnesium turnings and B-trichloroborazine in ether. The reaction once started, continues until the alkyl borazine is formed.

The infrared absorption spectra of isolated products consistently indicated the presence of ether which was difficult to remove (Fig. 2). The strong absorption bands characteristic of B-trimethylborazine (Ref. 9) were found at 6.75, 7.50, 11.1 and 14.0 microns; medium intensity bands were found at 2.95, 3.40, 9.25 microns and a weak band was found at 7.7 microns. However, a weak band reported at 12.24

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microns was completely absent. The peak at 8.8 microns is due to diethyl ether at a pressure of about three mm Hg.

The low yields encountered in the efforts to obtain B-trimethyl-borazine from the above mentioned reactions are difficult to explain. The logical assumptions are that the ethers (diethyl and THF) used were not completely dry and that there were large mechanical losses in separating the product from the mass of magnesium or lithium salts. Drying of the ethers with calcium hydride, use of a dry box and the fact that the solid discards from the reactor gave vigorous, exothermic reactions with water would indicate that anhydrous conditions were maintained in all the manipulations. Thus, mechanical losses seem to be the primary factor for the low yields. An alternate method of synthesis without the necessity of solvents is the pyrolysis of the ammonia complex of pyrophoric trimethyl boron at 300°C:

$$3(CH_3)_3B:NH_3 \longrightarrow B_3(CH_3)_3N_3H_3 + 6CH_4$$
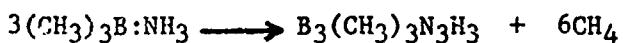
This method was not tried since the solution method appeared to be simpler without the necessity of special equipment not immediately available.

EXPERIMENTAL

Material and Experimental Method

1. Borazine

Borazine was obtained commercially (K & K Laboratories) as a liquid in screw cap vials. The liquid appeared "milky" from the decomposition and/or polymerization products. Therefore the material was immediately degassed, distilled into a glass ampoule, flame sealed and stored at -78°C until ready for use. Alternately, it was condensed into a glass bulb fitted with a stopcock and similarly stored; however, the possibility of channeling in the stopcock grease and thus exposure of borazine to the atmosphere necessitated short term storage when an ampoule with a stopcock was used. The original flask from which the borazine was fractionated contained a viscous residue which reacted vigorously and exothermically with water. The aqueous solution was basic and a strong amine-like odor, which disappeared on long standing, was evident. Evaporation of the water left white crystalline material which did not melt up to 200°C. A solution of this material with silver nitrate solution immediately formed a black precipitate of silver identified by its arc spectrum. The infrared spectrum of the material taken as a KBr pellet and its X-ray diffraction data are shown in Fig. 3 and Table 2 for reference.

Reactions of borazine were carried out on a conventional vacuum line with traps and manometers. Borazine from storage was expanded

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into a calibrated volume and measured. It was then condensed into a round bottom flask reactor previously evacuated. When solvents were used, these were first dried, degassed, then condensed into the reactor. Hydrogen chloride gas was obtained from a lecture bottle and measured in the calibrated volume of a vacuum line. Nitryl chloride reacts with mercury and therefore was approximated by condensing in a calibrated tubing and measuring the liquid. Solid products were handled in a dry box. Gaseous products were expanded into an infrared cell and analyzed.

2. Reaction of Borazine with Nitryl Chloride

Approximately 1.5 millimoles of borazine was condensed at -196°C in a 50 ml reactor. An excess of NO_2Cl was then condensed on the borazine. The liquid nitrogen was replaced by a -78°C cold bath. A violent explosion shattered the reactor and Dewar flask during the night. A similar experiment was made with the addition of THF as solvent. The mixture was thawed by slowly lowering the cold bath. A vigorous reaction ensued with color changes from orange to red to orange and finally a pale yellow solution remained. Some non-condensable gases at -196°C were present but were not analyzed.

3. Reaction of Borazine with Hydrogen Chloride

Borazine was expanded into a calibrated volume, measured and condensed in a 50 ml reactor. Gaseous hydrogen chloride in slight excess of a 3:1 stoichiometric ratio was condensed on the borazine. The reactor was then slowly brought to room temperature by lowering the cooling bath of liquid nitrogen. Unreacted gases were pumped off leaving the white solid complex.

4. B-Trichloroborazine

B-trichloroborazine was synthesized according to the method of Brown and Laubengayer (Ref. 10) but modified to accomodate a slow stream of boron trichloride from a lecture bottle.

$$3NH_4Cl + 3BCl_3 \longrightarrow B_3Cl_3N_3H_3 + 9HCl$$

Toluene (b.p. 100°C) and xylene (b.p. 140°C) were used as solvents in addition to chlorobenzene, but the best yield was obtained using the latter solvent. The result using cumene (b.p. 152°C) as solvent was entirely unsatisfactory. The solvent was removed under vacuum at room temperature and condensed in two cold traps before the mechanical pump. The evaporating solvent passed over a water cooled cold finger on which the product crystallized. However, much of the product was still lost by being carried over the cold finger.

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Since B-trichloroborazine rapidly reacts with moisture, extreme care was taken to maintain anhydrous conditions; however, no amount of care could present contact with air during some part of the operation. The difficulty was evident from the melting points of the products. Whereas B-trichloroborazine melts at 84°C, the products from most experiments did not melt till well over 100°C. Only a resublimed material obtained while observing the most rigid precautions melted in the range 84°C - 92°C. These rigid precautions could not be consistently observed and therefore the B-trichloroborazine was used without further purification.

Typically, five grams of ammonium chloride, previously dried at about 120°C, was weighed and transferred to a three-neck round bottom flask. Sufficient chlorobenzene to cover the solid was then introduced. The system was open to the atmosphere through a water-cooled condenser topped with a dry-ice condenser and connected to a drying tube of calcium sulfate. A lecture bottle of boron trichloride was connected to the system by way of a mercury bubble trap and a gas dispersion tube immersed in the chlorobenzene. The boron trichloride was introduced at a rate such that a drop would form at the tip of the dry-ice condenser every few seconds.

The mixture was agitated with a magnetic stirrer and was heated to about 155°C by means of a silicone oil bath around the reactor. Temperature was maintained for about eight hours. Progress of reaction could be seen by the decrease of ammonium chloride which never was entirely consumed. After termination of reaction, heating was continued for about one hour with a stream of nitrogen instead of boron trichloride in order to drive off unreacted boron trichloride as well as hydrogen chloride formed in the reaction.

The two side arms of the reactor were then capped and a water cooled cold-finger adapter (Fig. 4) was substituted for the reflux condenser. With cold tap water circulating through the cold-finger, the solvent was stripped from the reactor by means of a vacuum pump. Although B-trichloroborazine sublimes at room temperature, the solid material in the reactor was usually warmed to about 50°C to separate the product from unreacted ammonium chloride. The B-trichloroborazine condensed on the cold finger as long needles; however, much was lost either during solvent stripping or during the sublimation process.

When sublimation appeared to have ceased, the condenser was dried, the vacuum in the reactor was broken with nitrogen and the sublimation head removed into a dry box. The product was then scraped off the cold-finger into a screw cap bottle and weighed. The yield was generally better than 50 percent.

5. Reaction of B-Trichloroborazine

Typically, B-trichloroborazine was placed in a three-neck round bottom flask from a sample vial which was weighed before and after removal of sample. Minimum quantity of solvent was added to the flask. Reactions were either carried out in a plastic bag under constant nitrogen pressure or nitrogen was flushed through the reactor while a solution of the second components was added from a dropping funnel. After reaction, the flask was attached to the vacuum manifold and the components were separated by low temperature fractionation.

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TABLE 1

X-Ray Data for Borazine - 3HCl Complex

d	I**	d	I**
6.012	W	*1.931	MS
5.39	F	*1.731	M
4.31	VF	1.560	S
*3.86	S	*1.369	MW
3.18	M	*1.291	W
3.075	M	*1.245	MW
*2.728	VVS	*1.167	F
*2.229	MW	*1.118	VF
		*1.064	VVF

F = faint, VF = very faint, W = weak, M - medium

S = strong.

* These lines belong to cubic system: a = $3.89 \pm 0.05 \text{Å}$

** Intensities visually estimated from film.

TABLE 2

X-Ray Data for Product from Hydrolysis of
Residue in Borazine Container

d	I*	d	I*
6.06	26	2.93	10
5.57	22	2.84	12
4.64	10	2.54	8
3.56	26	2.36	7
3.40	22	2.18	9
3.39	23	2.06	8
3.29	12	2.01	7
3.20	80		

* Intensities based on peak heights from X-Ray diffractometer.
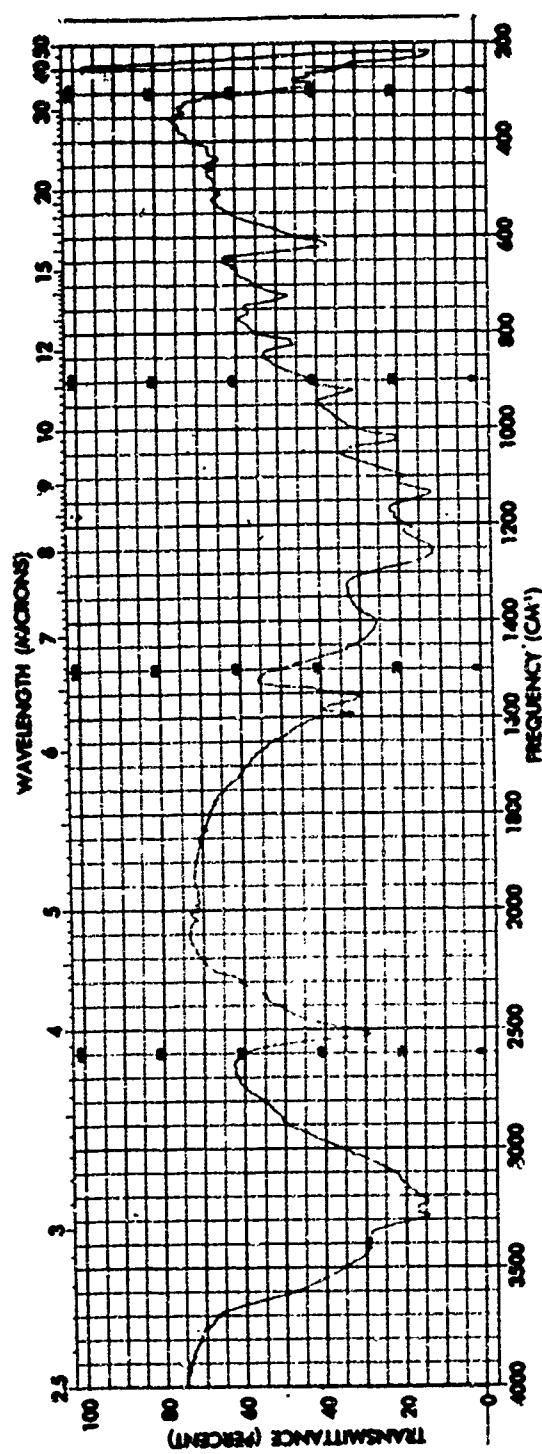
Fig. 1 Infrared Spectrum of Borazine-3HCl Complex
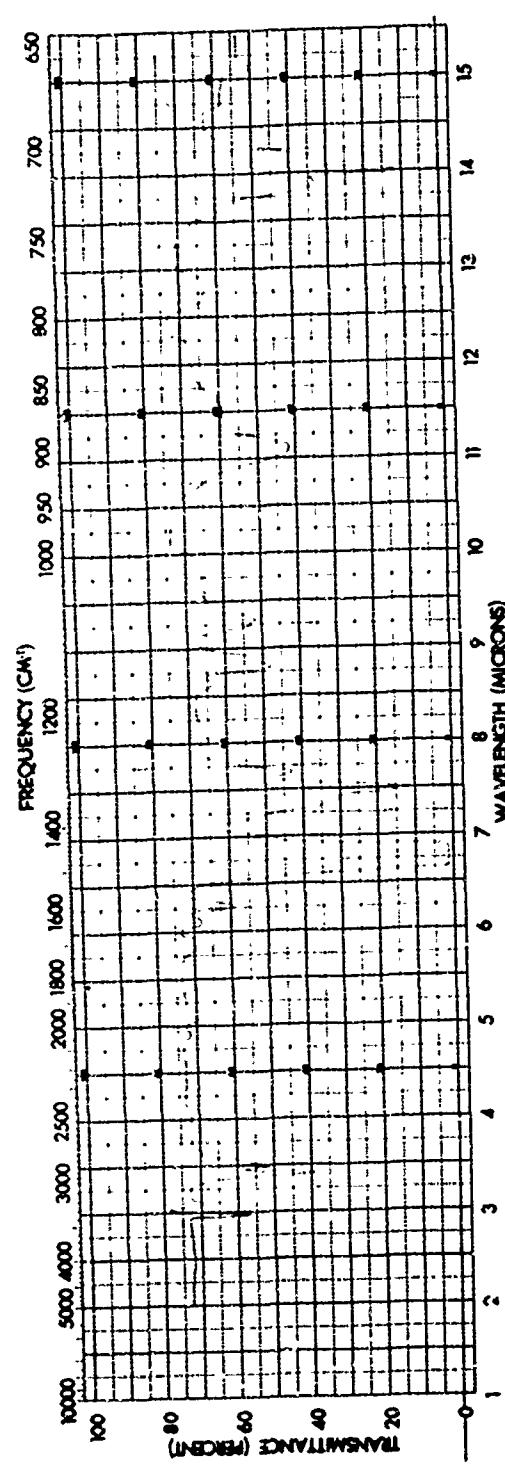
Fig. 2   Infrared Spectrum of Product from Reaction of
B-Trichloroborazine with Methyl Lithium

16
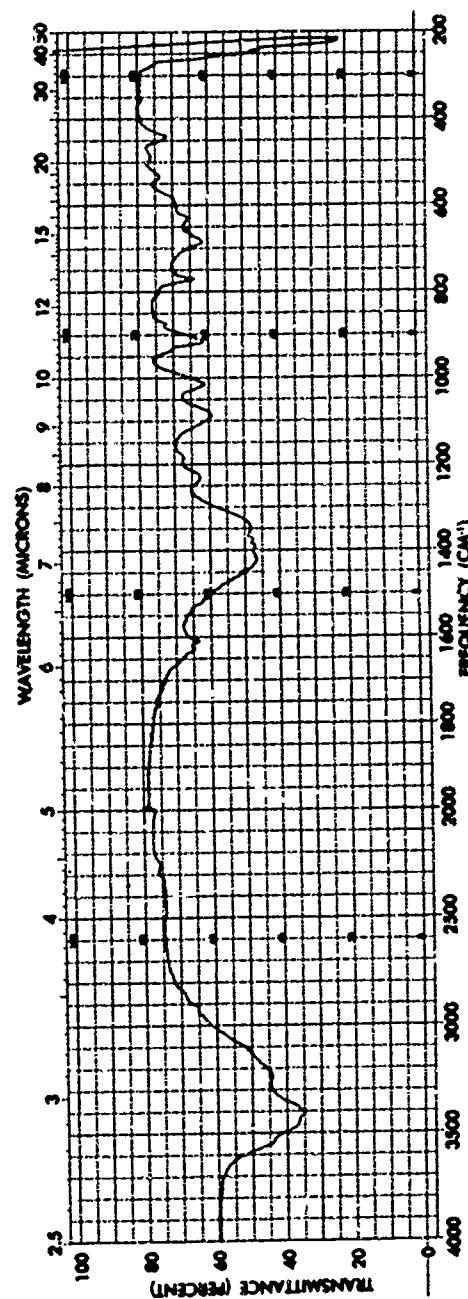
Fig. 3 Infrared Spectrum of Product from Hydrolysis of
Residue in Borazine Container

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H$_2$O In

H$_2$O Out

To vac pump

24/40 � Joint
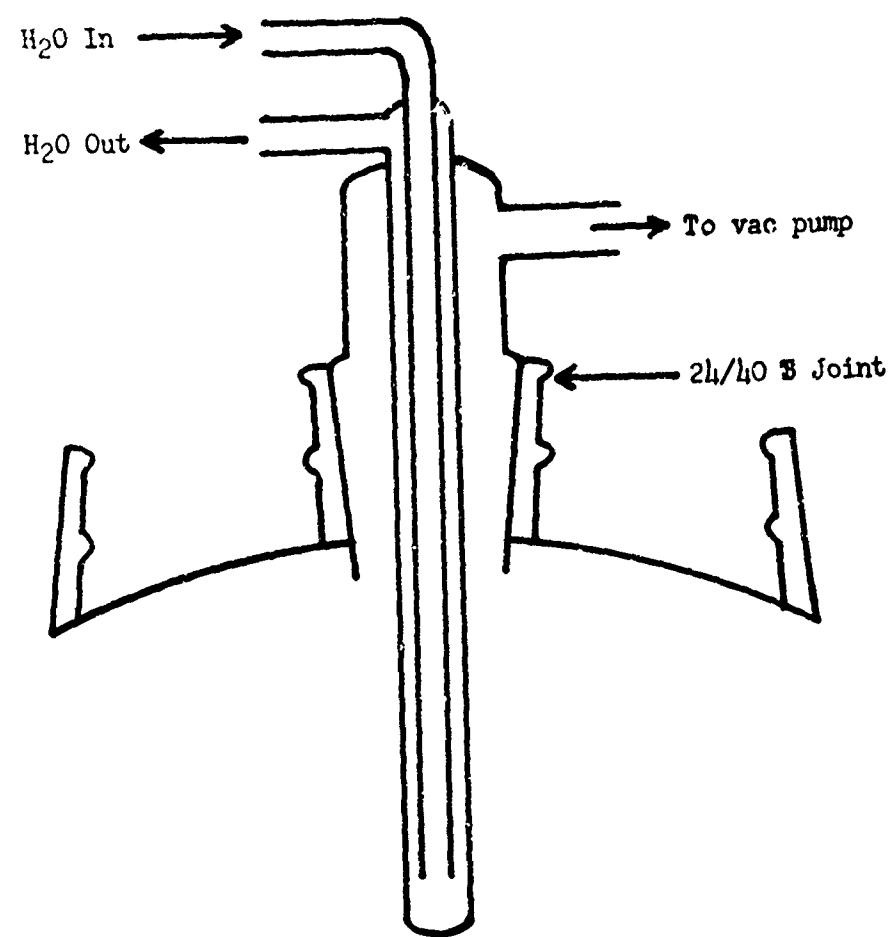
Fig. 4 Cold-Finger Condenser for Sublimable Material